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Depth measurement of doped semiconductors using the Hall technique

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A new method using the Hall technique to determine the change in surface layer thickness of doped semiconductors is presented. An equation to calculate the semiconductor thickness change has been determined by comparing the difference in Hall measured sheet carrier concentration and mobility before and after a change in surface layer thickness. Experiments were conducted using a wet chemical digital etch to remove *n*-type GaAs surface layers having an incremental etch depth control of approximately 15 Å in thickness, and the resulting thickness changes were calculated by the Hall technique and measured with a mechanical profilometer. This Hall measurement technique was able to measure changes in surface layer thickness of less than 100 Å, and the accuracy of this new technique compared favorably with mechanical profilometer measurements. The new Hall technique method provides accurate measurements of minute thickness changes, and is more accurate than mechanical profilometers for thickness changes less than 150 Å.

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INTRODUCTION

Shallow etching techniques are useful in fabricating quantum wires, gate recess etching of III–V semiconductor field effect transistors [such as metal-semiconductor field-effect transistors (MESFET), high electron mobility transistors (HEMT), and pseudomorphic high electron mobility transistors (*p*HEMT)], and for studying material and device properties of layers below the surface.^{1–5} Although mechanical profilometry and atomic force microscopy (AFM) are available, these methods have limitations. Mechanical profilometers can only accurately measure single edge etch depths of about 150 Å or larger. AFM is capable of measuring etch depths of atomic layer resolution and with atomic layer precision, but only across small areas (e.g., 100 μm²). Both mechanical profilometer and AFM require masking of the sample surface because the etch depth is determined by comparing the etched surface to the unetched surface. A new method has been developed that calculates the change in surface layer thickness based on differences in the Hall measured electrical parameters of carrier concentration, resistance, and mobility. This Hall technique does not require masking of the surface, and can be used with large surfaces (e.g., 1 cm²). Since the Hall measurement technique uses an electrical and not a mechanical measurement, errors due to mechanical vibration noises are eliminated and the resolution of the etch depth measurement is limited only by errors from the electrical measurement system. As an example, from accuracy and signal-to-noise considerations alone, the Hall technique can potentially resolve thickness changes of less than 1 Å in a 500-Å-thick GaAs layer doped at the 10¹⁸ cm⁻³ level; however, other factors may degrade this high resolution.

DEPTH MEASUREMENT USING THE HALL TECHNIQUE

Consider a semiconductor material of thickness *t* that is artificially segmented into *N* thin layers with depth Δ*z* such that (*t* = *N*Δ*z*), where Δ*z* is thin enough that the volume carrier concentration *n* and mobility μ do not vary appreciably within a given layer. It can be shown that the measured electrical parameters of sheet conductivity σ_□ and sheet Hall coefficient *R*_□ are given by^{6,7}

$$\sigma_{\square} = \sum_{i=1}^N \sigma_i \Delta z, \quad (1)$$

$$R_{\square} \sigma_{\square}^2 = \sum_{i=1}^N R_i \sigma_i^2 \Delta z, \quad (2)$$

where σ_{*i*} is the volume conductivity and *R*_{*i*} is the volume Hall coefficient of layer *i*. The measured quantities σ_□ and *R*_□ are related to mobility μ and sheet carrier concentration *n*_□ in the usual manner,

$$\sigma_{\square} = q \mu n_{\square}, \quad (3)$$

$$R_{\square} = 1/q n_{\square}. \quad (4)$$

The Hall scattering factor (*r*_H = *q n R*) is assumed to be unity, which is true for degenerate carriers (for example, when *n* > 10¹⁷ cm⁻³ in GaAs). The sheet and volume electrical quantities are related to the semiconductor thickness *t* by *n*_□ = *n t*, σ_□ = σ*t*, and *R*_□ = *R/t*. Note from Eqs. (3) and (4) that mobility (μ = σ_□*R*_□) can be calculated without a thickness measurement. The Hall technique is used to measure σ_□ and *R*_□ before (*j*) and after (*j* + 1) a change in thickness such that

$$\sigma_{\square j} - \sigma_{\square j+1} = \sum_{i=j}^N \sigma_i \Delta z - \sum_{i=j+1}^N \sigma_i \Delta z = \sigma_j \Delta z, \quad (5)$$

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TABLE I. Calculation of wafer carrier concentration and electrical thickness.

MBE-grown n layer		Hall measurement			Calculated results		
Wafer ID	Growth thickness t_m (Å)	Sheet resistance R_{\square} (Ω/\square)	Sheet carrier concentration n_{\square} (cm^{-2})	Mobility μ_n ($\text{cm}^2/\text{V s}$)	Volume carrier concentration n_0 (cm^{-3})	Electrical thickness t_e (Å)	Depletion thickness $(w_s + w_i)$ (Å)
1 ^a	350	1003.0	5.066×10^{12}	1230	5.978×10^{18}	84.7	265.3
2	700	168.1	2.599×10^{13}	1431	5.978×10^{18}	434.7	265.3
3 ^b	1000	90.4	5.065×10^{13}	1365	7.105×10^{18}	712.9	287.1
4	2000	38.8	1.217×10^{14}	1323	7.105×10^{18}	1712.9	287.1

^aWafers 1 and 2 grown back to back with the same MBE Si doping density flux.

^bWafers 3 and 4 grown back to back with the same MBE Si doping density flux.

$$\begin{aligned}
 (R_{\square}\sigma_{\square}^2)_{j-} - (R_{\square}\sigma_{\square}^2)_{j+1} &= \sum_{i=j}^N R_i \sigma_i^2 \Delta z - \sum_{i=j+1}^N R_i \sigma_i^2 \Delta z \\
 &= R_j \sigma_j^2 \Delta z = q n_j \mu_j^2 \Delta z. \quad (6)
 \end{aligned}$$

These equations are normally used to get n_j and μ_j [or $n(z)$ and $\mu(z)$] as a function of depth by measuring the semiconductor thickness change Δz at each step. However, if the volume carrier concentration and mobility are constant with depth and are known beforehand, then either Eq. (5) or (6) can be used to calculate the thickness change Δz due to etching.

The carrier concentration and mobility can be determined beforehand by comparing two separate semiconductor layers which are identical in every way except for metallurgical thickness t_m . The use of different layer thicknesses is necessary because the electrical thickness t_e is less than the metallurgical thickness t_m due to surface w_s and interface w_i depletion widths [see Eq. (9)]. If the carrier concentration and mobility are identical for both semiconductors (samples A and B) in the electrically conducting regions (t_{e_A} and t_{e_B}), then the carrier concentration can be determined by mathematically eliminating the depletion regions to give

$$n = \frac{n_{\square_B} - n_{\square_A}}{t_{m_B} - t_{m_A}} \quad (7)$$

where

$$w_s + w_i = \frac{(n_0 t_{m_A} - n_{\square_A})}{n_0} = \frac{(n_0 t_{m_B} - n_{\square_B})}{n_0}, \quad (8)$$

$$t_e = t_m - (w_s + w_i). \quad (9)$$

Since the measurement of mobility is independent of thickness, it should be the same for both samples A and B if the uniform material assumptions are correct. As seen in Table I the mobility is constant if the samples are thick enough, but begins to fall off as the semiconductor layer becomes thinner (350 Å in this case). One of the reasons for such a fall off is the fact that the depletion ‘‘walls’’ are not abrupt, but have a width approximately given by the Debye length (≈ 18 Å for $n = 5 \times 10^{18} \text{ cm}^{-3}$). Thus, for a very small electrical thickness ($t_e \approx 85$ Å), carrier concentration and mobility would be expected to vary over an appreciable portion of t_e . As observed, the mobility is lower because the decrease in carrier

concentration in the upper and lower Debye tails results in less screening of the ionized impurities. In other words, a larger fraction of the electrons see unscreened impurities and thus have lower mobilities.

From the experimental studies conducted for this work, it has been found that Eq. (5) seems to give the most consistent results for etch depth determination. Thus, solving Eq. (5) for the change in thickness in terms of $(\Delta z = t_1 - t_2)$, where $t_1 > t_2$) results in

$$\Delta z = \frac{\sigma_{\square_j} - \sigma_{\square_{j+1}}}{\sigma_j} = \frac{q \mu_j n_{\square_j} - q \mu_{j+1} n_{\square_{j+1}}}{q \mu_j n_j} \quad (10)$$

or

$$\Delta z = t_1 - t_2 = \frac{\sigma_{\square_1} - \sigma_{\square_2}}{\sigma_0} = \frac{\mu_1 n_{\square_1} - \mu_2 n_{\square_2}}{\mu_0 n_0}, \quad (11)$$

where n_0 and μ_0 are the initial volume carrier concentration and mobility of the semiconductor layer measured before any etching.

EXPERIMENTAL PROCEDURES AND RESULTS

A series of experiments was performed with GaAs to determine the accuracy of the Hall technique for calculating changes in thickness based on changes in Hall measured electrical parameters. The semiconductor material consisted of a thin layer (between 350 and 2000 Å) of n -type Si-doped ($\approx 5 \times 10^{18} \text{ cm}^{-3}$) GaAs grown on a semi-insulating GaAs substrate. Since the underlying substrate is semi-insulating, the electrical properties measured by the Hall technique are assumed to be attributed entirely to the n -type GaAs grown layer. The wafers consisted of two sets of molecular-beam-epitaxy (MBE) growths in which two wafers per set were grown sequentially using the same growth conditions and Si doping flux. The first set consisted of two wafers with identical Si doping density, one wafer with a 350-Å-thick n -GaAs layer and the other with a 700-Å-thick n -GaAs layer. The second set also consisted of two wafers with the same Si doping density, but with one wafer having a 1000-Å-thick n -GaAs layer and the other wafer having a 2000-Å-thick n -GaAs layer. Although all four wafers had approximately the same Si doping concentration, differences in Si doping flux between the first and second set occurred since the second set was grown several weeks after the first set.

TABLE II. Comparison between etch depths (change in thickness) from digital etching calculated from the Hall technique and measured by a mechanical profilometer.

No. of digital etch cycles	Hall technique		Mechanical profilometer	
	Average measured etch depth (Å)	Calculated etch rate (Å/cycle)	Average measured etch depth (Å)	Calculated etch rate (Å/cycle)
5	79	15.9
9	132	14.7
10	155	15.5	158	15.8
15	231	15.4	240	16.0
20	328	16.4	326	16.3
30	509	17.0	462	15.4

The *n*-GaAs layers were grown thin to maximize the change in measured Hall parameters from changes in layer thickness. The carrier concentration n_0 and electrically active thickness t_e of the wafers were calculated by comparing initial Hall measurements from both sets of wafers as given by Eqs. (7)–(9). The results of these calculations are presented in Table I.

Changes in material layer thickness can occur from either physical removal of the surface layer (e.g., reactive ion etching, ion milling, polishing, or grinding), or from chemical reactions that remove the surface layer (e.g., wet chemical etching or reactive ion etching) or change the electrical composition of the layer from conductive to nonconductive (e.g., oxidation or nitridation). For this work, a wet chemical digital etching technique was used to remove the *n*-GaAs surface layer so that changes in Hall measured parameters were due to changes in semiconductor thickness. The digital etching technique differs from standard wet chemical etching by separating the chemical reactions at the surface into two distinct processes.^{8–11} In digital etching, the first process step forms a surface film compound at a fixed depth due to the self-limiting nature of the first chemical reaction. The second chemical reaction selectively removes the newly formed surface film compound, but does not affect the unreacted GaAs region underneath. Therefore, the etch depth is dependent on the diffusion limited thickness of the surface film formed in the first step, and is relatively independent of the length of time the chemical solution is present on the surface. In this manner, a repeatable etch to fixed depth can be performed by repeating this two step etching process until the desired etch depth is reached. The advantage of the digital etching technique is that the self-limiting chemical reaction limits the etching process to shallow etch depths on the order of several atomic layers (from 5 to 30 Å).^{8–11}

For this work, the wet chemical digital etching technique consisted of using hydrogen peroxide (30% H₂O₂) to oxidize the GaAs surface to a fixed depth (first process step) and hydrochloric acid [HCl:H₂O (1:1)] to remove the GaAs-oxide layer (second process step).¹¹ The digital etching experiments were conducted on over 70 small samples cleaved from the four *n*-GaAs layers described earlier, with the number of digital etch cycles varied from 5 to 30. The Hall technique was used to measure the sheet concentration, sheet resistance, and mobility of the *n*-GaAs layer before and after each etch.^{6,7} For this work, it was convenient to use square

samples of about 6×6 mm² in size, with indium dots soldered on the corners and annealed at 425 °C for 3 min under a flowing inert gas. The thickness change from digital etching was calculated using Eq. (11) by comparing the change in Hall measured sheet resistance and mobility due to etching of the *n*-GaAs layer. For comparison with the calculated etch depths from Hall measurements, identical digital etching experiments were conducted on samples from the same four *n*-GaAs wafers, and their etch depths were measured directly using a mechanical profilometer. Different samples were needed since the mechanical profilometer requires masking of the sample to perform etch depth measurements between etched and unetched regions. The masking was accomplished using standard photolithographic techniques to open 100-μm-wide trenches for etching.

The average etch depth and etch rate values as calculated from the Hall technique and measured by a mechanical profilometer are presented in Table II and shown graphically in Fig. 1. The etch rate was calculated using

$$\text{digital etch rate } (\text{Å/cycle}) = \frac{\text{measured etch depth } (\text{Å})}{\text{number of digital etch cycles}} \quad (12)$$

As Table II shows, the Hall technique measured average semiconductor thickness changes ranging from 80 up to 500 Å. The etch depths calculated by the Hall technique are in agreement with measurements from the mechanical profilometer.

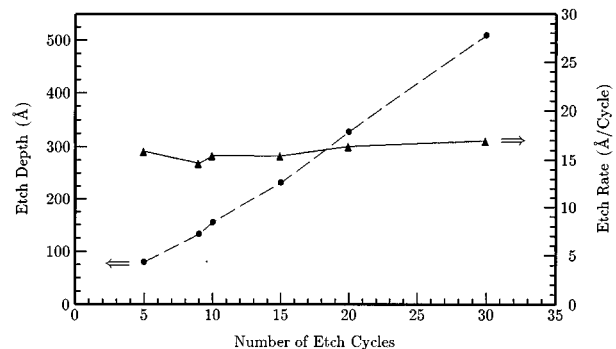


FIG. 1. Comparison of measured (●) etch depth (left-hand y axis) and calculated (▲) etch rate/digital etch cycle (right-hand y axis) as a function of the number of digital etch cycles performed. Experimental data used in this plot are from Hall measurements given in Table II.

meter in the thickness range from 150 to 500 Å, demonstrating the ability of the Hall technique to measure changes in semiconductor depth. Since the mechanical profilometer accurately measures etch depths of about 150 Å or larger, only etching experiments of ten or more digital etching cycles were performed for these samples. However, the Hall technique is capable of resolving much smaller thickness changes, so measurements down to five etch cycles (≈ 80 Å) were taken. Even without mechanical profilometer measurements to corroborate these smaller etch depths, a unique property of digital etching allows for comparison of the Hall measurement technique with larger etch depth measurements. That is, with digital etching the digital etch rate remains independent of the number of digital etch cycles performed.⁸⁻¹¹ So, even though increasing the number of digital etch cycles produces larger etch depths, the digital etch rate as calculated by Eq. (12) remains constant. In digital etching the final etch depth is determined by the number of digital etch cycles performed, instead of by the etch time as in standard etching. Therefore, the amount of material etched is always a multiple of the single cycle etch depth (digital etch rate). Figure 1 graphically displays the linear relationship between the etch depth and the number of etch cycles, while also showing the independent (constant) relationship between the digital etch rate and the number of etch steps. Since the calculated digital etch rate for 5 \times and 9 \times digital etch cycles as measured by the Hall technique is equal to the digital etch rate for larger etch depths, it can be asserted that accurate measurement of etch depth changes of less than 100 Å is possible using the Hall technique.

The resolution and repeatability of the Hall technique was tested by repeating Hall measurements on several *n*-GaAs samples over time without etching to see if the measured Hall parameters remained constant. Measurement of the Hall electrical parameters during the same day resulted in negligible changes from 0.05 Å to a maximum of 1.7 Å in calculated thickness change, with the average change in thickness of the samples being 0.38 Å. If the measurement of the Hall parameters was performed on succeeding days, then the average change in thickness increased to 3.94 Å after 1 day from the original Hall measurement and increased further to 5.30 Å after 2 days. Soaking the *n*-GaAs samples for 2 min in HCl:H₂O (1:1) 1 day after the initial Hall measurements resulted in a calculated change in thickness of 6.63 Å, which increased to 8.22 Å when soaked for 2 min in HCl:H₂O (1:1) 2 days after initial Hall measurements. The consistency of the Hall measurements made in the same day show good repeatability, and the Hall measurements performed on successive days show the resolution of this mea-

surement technique in calculating the change in thickness that is expected from native GaAs-oxide formation from the exposure to air.¹²⁻¹⁴ Although better methods of measuring this native oxide layer exist (i.e., ellipsometry), and the Hall measurement system may not be accurate with absolute thickness changes in the 1-10 Å thickness range, the results indicate thickness changes that can be expected from native oxide growth on a GaAs surface.

CONCLUSIONS

This work demonstrates that the Hall technique can accurately measure small changes (<100 Å) in doped semiconductor material thickness. Because this technique does not require masking and measures the average changes in etch depth over large areas (e.g., 1 cm²), it is useful where shallow etch depths are investigated. The accuracy and resolution of the Hall technique makes this an ideal measurement tool to use with shallow etching techniques such as digital etching, which removes several atomic layers of material per etch cycle.

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